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QUARTZ CRYSTAL MICROBALANCE CONTAMINATION MONITORS ON SKYLAB — A QUICK-LOOK ANALYSIS

By R. Naumann, W. Moore, D. Nisen, W. Russell, and P. Tashbar Space Sciences Laboratory

June 8, 1973

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INTRODUCTION

Because of a lack of understanding of the behavior of contaminants from various sources and because of the extreme susceptibility of some of the Skylab and Apollo Telescope Mount (ATM) experiments to thin contaminating films, six quartz crystal microbalances (QCM's) were included on the Skylab mission to provide real-time contamination deposition measurements for the purpose of making operational decisions. Also, these measurements will help to resolve some of the uncertainties in our present capability to predict the behavior of contamination from various sources in the space environment.

DESCRIPTION

The microbalances consist of two A-T cut 10-MHz crystals mounted back to back to minimize temperature differences (Fig. 1). The forward crystal is the sensing crystal, which responds to any addition of mass that may be deposited on it by a decrease in its resonance frequency. The rear crystal is protected against material deposition and serves as a reference crystal. The two crystals are driven by independent oscillator circuits in which the crystal itself is the frequency controlling element. The two output frequencies are mixed by a dual-gate MOSFET. The sensing crystal is biased approximately 1 kHz below the reference crystal; therefore, any mass deposition will result in an increase in beat frequency.

Quartz crystals are also sensitive to temperature changes. Thermal effects are minimized by selecting an A-T cut that has a low thermal sensitivity over the range of operating temperatures and by carefully matching the thermal response curves of the sensing and reference crystals. Locating the crystals back to back with a very small separation between them minimizes temperature differences. When these techniques are used, thermal variations can be held to a few tens of hertz over a temperature range from +90°C to -45°C. When

the sensing crystal is exposed to direct sunlight, a thermal gradient is present, which causes a drop of approximately 200 Hz in beat frequency. The thermal mass of the crystals is so small that this drop occurs in 10 sec. Therefore, as the spacecraft moves in and out of the shadow, a QCM looking at the sun will produce an almost square wave response. The deposition information is obtained by comparing readings taken from successive orbits under the same sunlight conditions.

The sensitivity of the system can be calculated from theoretical considerations by treating the deposited mass as an increase in crystal thickness. This results in a sensitivity of $2.257 \times 10^8 \, \text{Hz/}\mu\text{gm/cm}^2$ for a 10-MHz A-T crystal vibrating in the shear mode. The only deviation from the theoretical value of sensitivity comes from the assumption that the deposited material moves with the quartz surface. For fairly thick liquid films, only the first several monolayers will actually be sensed. The dissipation in the remainder of the film damps the crystal and causes it to cease oscillation after approximately 30 $\mu\text{g/cm}^2$ have deposited. For more nearly solid films, depositions up to $60 \, \mu\text{g/cm}^2$ can be measured before saturation occurs.

The crystals were calibrated by exposing them to a flux of DC-705 vacuum pump oil in a vacuum chamber. The thickness of the film was measured in situ by an ellipsometer, and the mass was calculated from the density. The sensitivity was obtained by dividing the observed frequency shift by the measured surface deposition. The results are shown in Table 1.

Film Thickness Sensitivity, $\times 10^8$ Deposition Δ F (**Ă**) $(\mu g/cm^2)$ (Hz) $(Hz/\mu g/cm^2)$ 147 1.57 330 2.11 226 581 2.41 2.42 739 7.88 1736 2.35 2.257 (theoretical value)

TABLE 1. QCM CALIBRATION RESULTS

The measured sensitivity is very consistent with the theoretical value for mass depositions up to at least 7.88 $\mu g/cm^2$. Electronic and thermal stability are such that the fluctuations are less than 1 Hz. The sensitive area is 0.317 cm². Therefore, the unit is actually capable of measuring mass changes on the order of nanograms.

Two QCM's (designated HCO and NRL-B) are mounted on the ATM sunshield looking along the +z axis (Fig. 2). The crystals are slightly recessed and have a field of view of 4.14 steradians (70-deg half-cone angle). There is no part of the spacecraft in the direct field of view of these units; therefore, their primary function is to monitor the return flux of contamination molecules that could enter the ATM aperture doors. They will also monitor the effects of docking and other orbital operations, such as EVA, on the ATM experiments.

Four QCM's are mounted on a truss below the Multiple Docking Adapter (MDA) in the vicinity of the Earth Resources Experiment Package (EREP) experiments. These units have a 1.59-steradian field of view (42.5-deg halfcone angle). Two of these units look in the -z direction. One unit is designated ZAM and operates at the ambient temperature of the truss assembly (0 to -23°C). The other unit, designated Z+50, is insulated to retain some of its internal heat in an attempt to elevate its temperature to that of the S-190 window, which is controlled at 10°C (50°F). These units have no part of the spacecraft in their field of view although, as may be seen in Figure 3, the ZAM unit does have the wire and connector from the CSM unit in its field and the Z+50 unit can see the face of the OWS unit. The unit designated CSM looks along the +x axis toward the Command/Service Module (CSM), and the unit designated OWS looks along the -x axis toward the Orbital Workshop (OWS) forward dome which is covered by the meteoroid curtain. Figure 4 shows the location of these units on the Skylab cluster. The primary purpose of these units is to monitor the environment in the vicinity of the EREP experiments and to assess the contamination associated with docking, molecular-sieve operation, and other spacecraft functions.

To be compatible with the Skylab data system, the frequency must be converted to a 0- to 5-volt signal. This is accomplished by means of an F/V convertor which converts the sine wave output to a train of precision pulses whose repetition rate is the sine wave frequency. This train is applied to a 'leaky bucket' integrator consisting of an R-C network with a bleed resistor across the capacitor. The limiting factor on the sensitivity in the system is the voltage resolution in the Skylab telemetry. For the ATM units, the ATM telemetry system provides a 10-bit word which has a voltage resolution of 4.8 mV. The F/V conversion is 1625 Hz/volt; therefore, the mass resolution is $\pm 0.034~\mu g/cm^2$. The EREP units use the Airlock Module (AM) telemetry system which is limited to 8 bits. Therefore, a range extender was added which multiplies the output voltage by 8 and subtracts multiples of 5 volts until the output is in the 0- to 5-volt range. This is equivalent to an 11-bit word and provides $\pm 0.017~\mu g/cm^2$ mass resolution.

RESULTS

The four EREP QCM's were tested for liveness after installation by spraying them with Freon. Since Freon is an excellent solvent for oil, this left a slight oil film on the crystals (6.00 $\mu g/cm^2$ on the CSM unit, 2.91 $\mu g/cm^2$ on the OWS unit, 2.97 $\mu g/cm^2$ on the ZAM unit, and 19.38 $\mu g/cm^2$ on the Z+50 unit. Because of this oil film, the CSM and Z+50 units were cleaned on the pad just prior to launch. Cleaning lowered the CSM unit to 0.70 $\mu g/cm^2$ below the original value and lowered the Z+50 unit to 0.14 $\mu g/cm^2$ below its initial value.

For convenience, a zero mass deposition reference level is chosen to represent a clean crystal. This choice is somewhat arbitrary since there is no way of knowing when the crystal is absolutely clean. Because all the crystals exhibited an initial decline for the first 1.5 hours after launch, the readings at day 134:19:00 were selected to represent clean crystals, and all mass additions are referenced to these values. Figure 5 indicates a history of the EREP units prior to launch and during the first day after launch. Notice that the two units that had not been cleaned (OWS and ZAM) lost their oil residue rapidly after orbital insertion, whereas the cleaned units (Z+50 and CSM) lost practically no mass during insertion. All units appeared to pick up some material from T + 90 min to T + 240 min, when it begins to come off. Whether this represents real deposition or some other effect, such as the reference crystals cleaning up, cannot be determined until a more detailed analysis is performed, but the latter is believed probable. Both the crystals initially have several monolayers of atmospheric molecules and water vapor sorbed on their surface. The sensing crystal has a much higher pumping speed because it is directly exposed to space. This would result in a rapid drop followed by a slow rise in indicated mass.

The long-term behavior can be seen in Figure 6. The CSM unit began picking up deposition as soon as the cluster was placed at a 50-deg sun angle. Presumably this allowed sunlight to fall on a component in the field of view, such as the L-band antenna, which stimulates outgassing. The rate was observed to be 0.21 $\mu g/cm^2/day$. The OWS unit collects an almost steady rate of 0.34 $\mu g/cm^2/day$. This is presumably outgassing material from the meteoroid curtain over the forward OWS dome and from the battery pack and the X-band antenna. The ZAM unit was collecting at the rate of 0.03 $\mu g/cm^2/day$ prior to docking, and the Z+50 unit slowly cleaned up, leveling out at 0.4 $\mu g/cm^2$ below the reference level.

The view factor for the OWS unit is estimated to be 0.46. This factor multiplied by the outgassing rate of the surfaces in the field of view gives the impingement rate. Assuming that everything that hits sticks, the average

outgassing rate would have to be at least 0.74 $\mu g/cm^2/day$ or 8.5 \times 10⁻¹² $\mu g/cm^2/sec$. This is not an unrealistic outgassing rate for paints and other nonmetallic materials at temperatures above 0°C, but preliminary indications are that all the surfaces in the field of view are substantially colder than this. Therefore, the source of this material has not yet been determined.

The ATM QCM's (Fig. 7) were first turned on at 32 min after launch. Therefore, the initial outgassing could not be seen. Consequently, the last readings before lift-off were taken as the reference. When the units first received power, they both indicated 0.24 $\mu g/cm^2$ above the reference value. Again, this could be a result of thermal shifts, the reference crystals cleaning up, or some contamination that may have occurred during launch.

As these units were exposed to the sun, a gradual loss of mass was observed. At present, the HCO unit has lost a total of 1.3 $\mu g/cm^2$ (130 Å of $\rho=1$ gm/cm³), and the NRL-B unit has lost 2.4 $\mu g/cm^2$ (240 Å). A similar effect was observed during testing of these units during the thermal vacuum test TV-3 in the Johnson Space Center (JSC) Chamber A under solar simulation. It was originally thought that this mass decrease resulted from outgasing products from the S-13G paint on the QCM mount that found their way through the vent hole and deposited on the reference crystal. Consequently, the vent on the flight unit was altered to preclude this possibility. It now appears that the observed result must be associated with actual mass loss from the sensing crystal, or from migration of contaminants between the two crystals to the cooler reference crystal. Additional tests must be performed to determine which effect is responsible for the observed behavior.

It is interesting to investigate the contamination effects of rendezvous and docking. Figure 8 shows the voltage readings from the EREP QCM's during rendezvous. Note that the OWS, ZAM, and Z+50 units respond as the Apollo Command Module approaches from the rear. The CSM unit responds two minutes later as the Apollo moves in front of the Skylab. Also note that the minor fluctuations in the response of the Z+50 and ZAM units are almost perfectly correlated. Therefore, these fluctuations must be caused by actual fluctuations in the local environment rather than instrumentational effects.

The ATM QCM's also respond at 20:40. The fact that the NRL-B-unit does not appear to respond at 20:40 is a peculiarity of the ground data retrieval system. This system records the same value unless a different signal is obtained. At 20:40 the station was just losing a signal. The increase was noted on the real-time telemetry but was not picked up on the data retrieval system.

The complete analysis of the effects of rendezvous must wait for detailed trajectory information and processed data tapes. It is known that the CSM

passed under and to the front and then executed a fly-around inspection, but it is not yet clear exactly where the CSM was at 20:40. The observed effects could be the result of Reaction Control System (RCS) firings. It is also possible that the shift of the ATM crystals at 20:40 could be the result of an attitude maneuver that took place at that time. Since the QCM's were in the sun, a change in sun angle would produce a rapid shift of a few hertz.

The contamination associated with the rendezvous and fly-around ranged from 0.14 $\mu g/cm^2$ on the OWS QCM, which was partially shadowed by the OWS, to 0.556 $\mu g/cm^2$ for the CSM QCM, which has a much greater exposure to the CSM.

The soft dock maneuver resulted in a mass increase of $2.33 \,\mu\text{g/cm}^2$ and a decay rate of $0.162 \,\mu\text{g/cm}^2/\text{hr}$. The OWS QCM showed an increase of $0.108 \,\mu\text{g/cm}^2$ from the docking, presumably from RCS plume reflection from the OWS forward dome. The other QCM's collected only $0.09 \,\mu\text{g/cm}^2$.

Figure 9 shows the EREP QCM raw data during the Standup Extravehicular Activity (SEVA) and repeated attempts at hard dock. A large amount of RCS propellant was consumed during this time. The CSM QCM had accumulated a total of at least $16.7 \,\mu\text{g/cm}^2$, $12.93 \,\mu\text{g/cm}^2$ during SEVA and the various attempts at docking. A gain $(0.323 \,\mu\text{g/cm}^2)$ was seen on the OWS QCM before and after the SEVA and docking sequence. Note the slight increase and subsequent decay of the -z axis QCM's during each docking attempt. This may be caused by material scattered from the plume or material from the plume undergoing multiple reflections. In any event, it appears to leave rapidly.

The material deposited on the CSM QCM has a maximum decay rate of $-6.15\,\mu\mathrm{g/cm^2/hr}$. For a material with a molecular weight of 100, and $\rho=1~\mathrm{g/cm^3}$, a monolayer has a mass of $5.5\times10^{-8}~\mathrm{g}$. An evaporation rate of $6.15\,\mu\mathrm{g/cm^2/hr}$ or $1.708\times10^{-9}~\mathrm{g/cm^2/sec}$ would require a surface stay time of 32 sec. For a surface temperature or 273°K (the QCM had a temperature ranging from $-15^{\circ}\mathrm{C}$ to $+8^{\circ}\mathrm{C}$ during this time), the heat of adsorption must be approximately 18 to 23 Kcal/mole and the vapor pressure must be 4.8×10^{-8} torr.

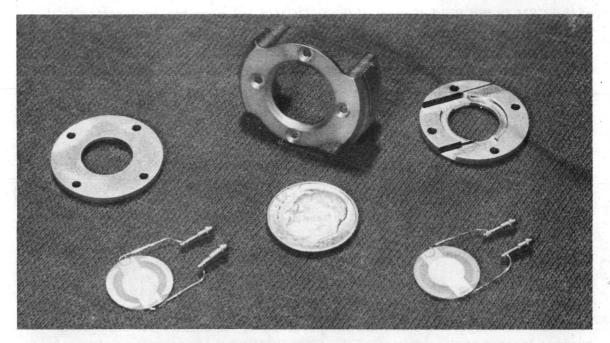
After docking, the CSM QCM continues to decline, leveling out at day 152. The OWS QCM continues to collect at nearly the same rate as it has throughout the mission. The peculiar thing is that the ZAM has increased its rate from 0.097 $\mu g/cm^2/day$ to 0.216 $\mu g/cm^2/day$, and the Z+50, which was collecting nothing before the dock (day 145) is now (day 147) collecting at the rate of 0.097 $\mu g/cm^2/day$. This is not well understood since there is no part

of the CSM in the field of view of these QCM's. A possible explanation is that RCS material has deposited on the face of the OWS QCM and the connector of the CSM QCM, which are in the fields of view of the Z+50 and ZAM QCM's, respectively, and is slowly evaporating onto the -z-facing units. More detailed analysis is necessary to confirm this possibility.

CONCLUSIONS

The QCM's are operating very much as expected and are providing excellent information for the first time on the behavior of contamination in the vicinity of a large manned spacecraft. From the preliminary data, the following tentative conclusions can be drawn about the contamination problem:

- 1. Surfaces that have portions of the spacecraft in their field of view collect a considerable amount of contamination. The amount depends on the view and its temperature and the temperature of the collector. An optical surface located at the position of the OWS QCM would have collected 700 $\mathring{\rm A}$ of contamination at this time. This would produce significant degradation of an optical surface operating in the ultraviolet and measurable degradation in the visible region. The amount of contamination is surprising considering the temperatures of the materials in the field of view.
- 2. It appears that surfaces can be effectively protected by shadow shielding or by locating them in such a manner that no contamination source is in their field of view. There was no evidence of material returning to the spacecraft and depositing on surfaces prior to CSM docking. However, the accumulation of material on the ZAM and Z+50 QCM's since CSM docking is not yet completely understood.
- 3. The use of RCS thrusters will produce considerable contamination on surfaces exposed to their plume. However, there appears to be little or no material scattered from the plume so that shielding from direct exposure to the plume appears to be an effective protective method.
- 4. Surfaces exposed to the sun appear to lose mass. This may be caused by the loss of adsorbed material previously pinned under the surface coating that migrates through the coating under the action of solar flux. More investigation of this phenomenon is required before a conclusion can be reached.



a. Crystal assembly showing the crystals, crystal holders, and crystal mounting assembly.



b. Assembled crystal holder and electronics which include a regulated power supply, two oscillators, mixer, F/V convertor, and EMI suppression filters.

Figure 1. Quartz crystal microbalance.

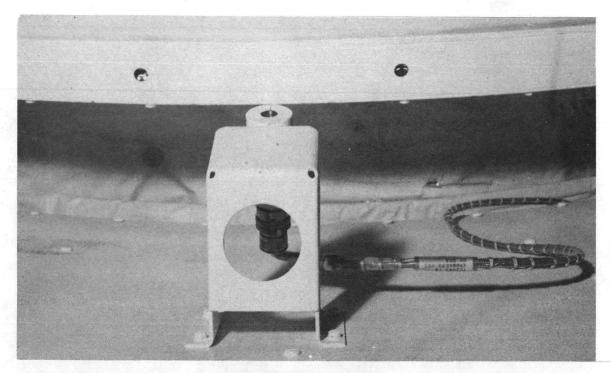


Figure 2. QCM mounted on ATM sunshield.

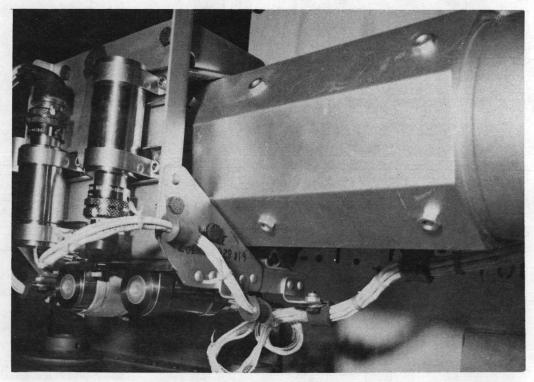


Figure 3. EREP QCM assembly. (The vehicle X-axis is to the right and -Z-axis is up.)

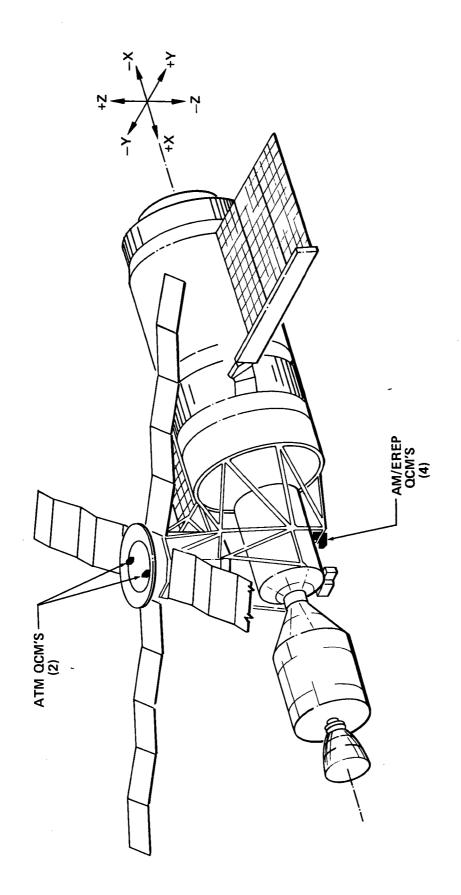


Figure 4. Location of QCM's on Skylab.

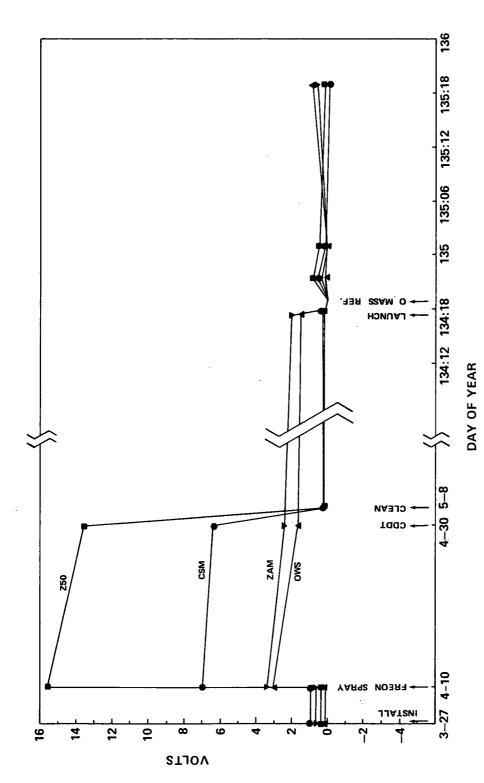


Figure 5. History of EREP units prior to and during orbital insertion. (Times prior to lift-off are not to scale but serve to indicate events that occurred. Note that the units that had been clean prior to launch did not change appreciably as the ambient pressure dropped, whereas the units that had not been cleaned exhibited rapid cleaning as the ambient pressure declined. The readings at 19:00 were arbitrarily selected as the zero reference to mass change.)

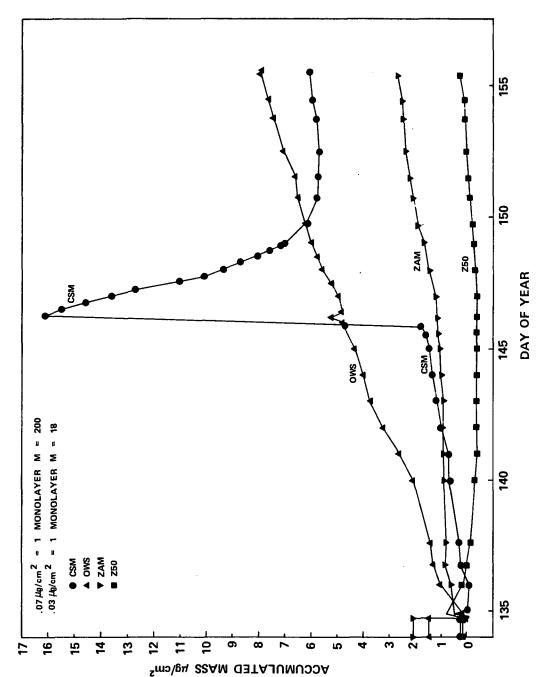


Figure 6. Long-term mass deposition measurements on the EREP QCM's. (Note the effect of docking on the CSM unit.)

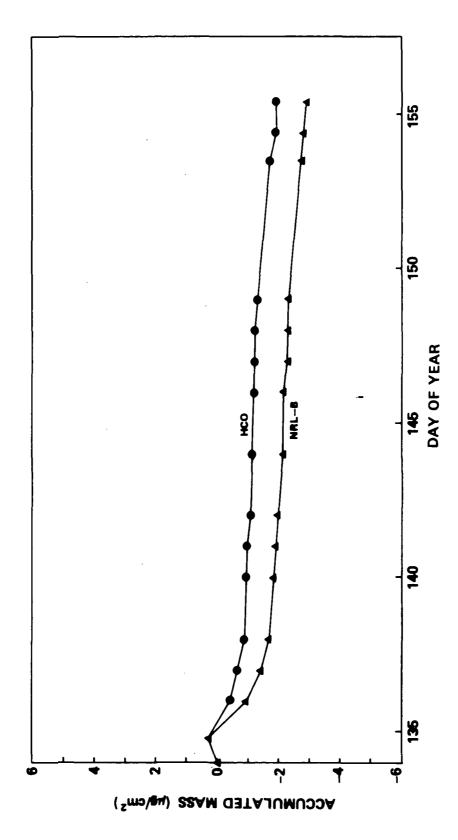


Figure 7. Long-term mass deposition measurements from the ATM QCM's. (After orbital insertion, the units continue to clean under the solar-vacuum environment.)

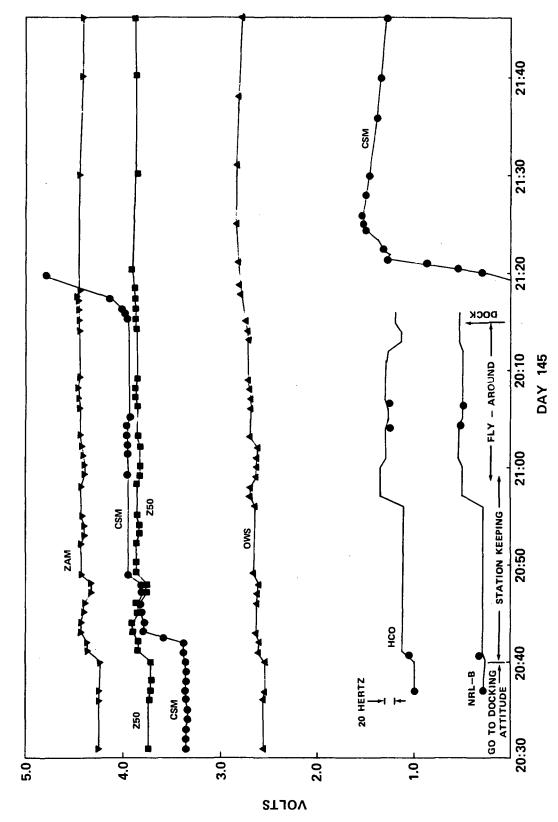


Figure 8. High time resolution of the EREP QCM raw data during first rendezvous and soft-dock.

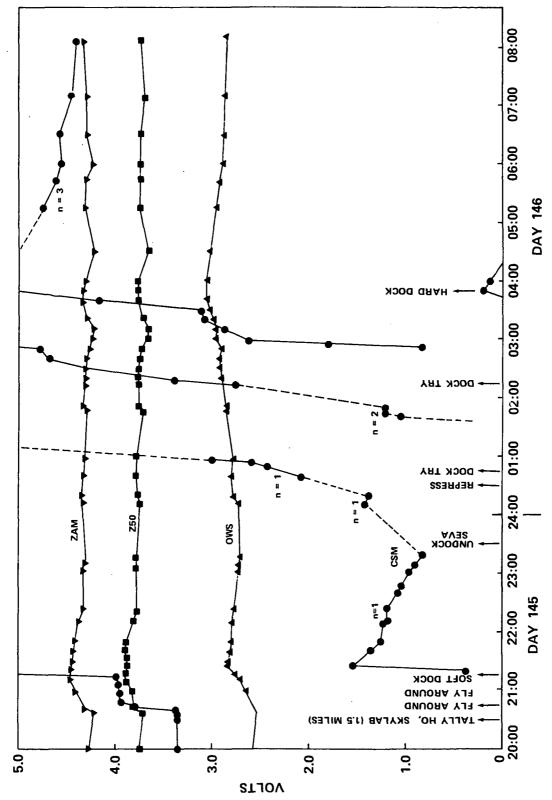


Figure 9. Detail of EREP QCM raw data during SEVA and docking attempts.

APPROVAL

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The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

CHARLES A. LUNDQUIST

Director, Space Sciences Laboratory

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